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Standardization activity in the evaluation of moisture content

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ABSTRACT

This review paper comments the international standards to measure the moisture content in building materials, i.e. EN 772-10:1999; EN 13183-1:2002; EN 13183-2:2002; EN 13183-3:2005; EN 1428:2012; EN-ISO 11461: 2014; EN-ISO 15512:2014; ISO 11465:1993; ISO 12570: 2013; ISO 16979:2003 and ISO 760:1978. The above standards do apply to new building materials, with standardized composition and shape, in satisfactory state of conservation, without sampling restrictions. If they are applied to aged and deteriorated materials, as in the field of cultural heritage, the results may be misleading. The paper discusses the difference between 'moisture content' and 'water content' and the various problems met with cultural heritage materials, e.g. ethical problem when test specimens are needed; the biased response when wood was attacked by moulds or insect tunnelling, or was impregnated with oil, wax or preservatives; or when masonry contains soluble salts or subsurface discontinuities. The most recent, comprehensive standard is presented, i.e. EN16682 (2017) '*Conservation of cultural heritage – Methods of measurement of moisture content, or water content, in materials constituting immovable cultural heritage*' that considers all existing methods and discusses pros and cons of each of them in relation with the real world of aged and deteriorated materials.

1. Introduction

This review paper will introduce the topic of Modhima and comment the European and international standards to measure the moisture content in building materials. They have been produced by the European Committee for Standardization (CEN), that is an association that brings together the National Standardization Bodies of 34 European countries and by the International Organization for Standardization (ISO), that is a worldwide federation of national standards bodies (ISO member bodies). CEN standards are abbreviated EN owing for European Norms. The work of preparing ISO or EN Standards is normally carried out through Technical Committees with specific competence on the topic. A number of standards produced by ISO have been adopted by CEN and appear under the two headings EN-ISO. Some specific standards have been produced in association between ISO and CEN.

The EN standards [1–5], the ISO standards [6–10], and the EN-ISO standards [11,12] provide a precise operative protocol and describe the instrumentation needed for some selected methods to measure the moisture content. Each standard deals with only one method that is specified in the title e.g. gravimetric determination, chemical titration, azeotropic distillation, electrical resistance measurement, capacitance measurement, etc. The standardization by single subject may be confusing because all standards, and therefore all methods, are apparently at the same level, and the user lacks a comprehensive framework to assist him in the choice of the most convenient one for every particular case study. In addition, not all the existing methods have been standardized.

In this review, the standards [1–12] for normal materials will be considered and commented for uncertainties or inappropriate use, i.e. when the test material is deteriorated. The difficulties, and the misleading results, that the user may have if a standard for normal materials [1–12] is applied to cultural heritage materials will be also considered.

In 2017, CEN, Technical Committee 346, has produced a comprehensive standard, EN 16682 [13] specific for cultural heritage, but usable for most materials, which constitutes an authoritative guideline to choose and use all existing methods and advices about misuse and misinterpretation of readings. This paper will outline the benefits that the user may gain from this holistic standard as well as some outstanding problems.

In the following, for the sake of clarity, when standards are introduced, they will be indicated with their number, full title and bibliographic reference.

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2. Materials and methods, with discussion

The various methods are here shortly presented, followed by a comment about potential flaws in the case of misuse, e.g. application to non-recommended materials, or materials that have been altered over time, or are in bad condition of conservation.

2.1. Gravimetric method

A number of EN and ISO standards concern the so-called gravimetric method. It consists in determining the mass of a moist test specimen of the material, drying it to constant mass, determining the new mass, calculating the ratio of the loss in mass during drying to the mass after it has been dried . The result should be expressed in % of the dry test specimen.

The following standards refer to it:

- EN 13183-1. *Moisture content of a piece of sawn timber – Part 1: Determination by oven dry method* [1].
- EN 772-10. *Methods of test for masonry units – Part 10: Determination of moisture content of calcium silicate and autoclaved aerated concrete units* [5].
- ISO 11465. *Soil quality – Determination of dry matter and water content on a mass basis – Gravimetric method* [6].
- ISO 12570. *Hygrothermal performance of building materials and products – Determination of moisture content by drying at elevated temperature* [7].
- ISO 12571. *Hygrothermal performance of building materials and products – Determination of hygroscopic sorption properties* [8].
- ISO 16979. *Wood-based panels – Determination of moisture content* [9].
- EN-ISO 11461. *Soil quality – Determination of soil water content as a volume fraction using coring sleeves – Gravimetric method* [11].
- EN 16682. *Conservation of cultural heritage – Methods of measurement of moisture content, or water content, in materials constituting immovable cultural heritage* [13].

Discussion. The gravimetric method is very popular, accurate and absolute. 'Absolute' means a method whose readings can be expressed in terms of the International System of units (SI), i.e. grams, corresponding to the weight of the moist and dry specimen. When the reading is expressed in %, the SI formally disappear, but they have been essential in determining the final reading.

The standards [1,5–9,11] describe how to proceed, but they do not establish whether this method should be preferable to others. As opposed, the American standard ASTM D4442 '*Standard Test Methods for Direct Moisture Content Measurement of Wood and Wood-Based Materials*' [14] specifies that the gravimetric method should be considered "*primary*" method, and others should be considered "*secondary*" ones. Similarly, EN 16682 [13] establishes priorities in methods, and establishes that the gravimetric method and the Karl Fisher Titration should be used as primary reference.

In the gravimetric method, sampling is made on the site, but weighing and drying are performed in the laboratory. In the field of cultural heritage, sampling constitutes an ethical problem because it is a destructive methodology that should not be applied, except for exceptional, well justified cases, that should be under the supervision and responsibility of the competent authority. A practical problem with cultural heritage materials is the lack of homogeneity in the material, and specimens taken in different parts may provide different results and it is not possible to take benefit of the average of several specimens to avoid sampling damage. In particular, in order to minimize this damage, specimens are generally extracted from less representative parts, e.g. near the borders, in already damaged areas, or their depth is limited to a surface layer. The risk for scarce representativeness cannot be avoided increasing the number and dimension of the test specimens, as discussed.

This method is simple and objective. It may provide reliable results, except when the material includes components not resistant to oven temperature. For instance, if the material has been treated with, or includes oils, waxes, resins, or other substances that at oven temperature become volatile organic compounds (VOC) and are outgassed, the mass difference of the specimen after drying is due to the sum of the two contributions, i.e. water and VOC. Therefore, the gravimetric oven drying method overestimates the moisture content when the test specimen includes substances that may be outgassed. In any case, the official definition of "*moisture content*" is based on this method and is potentially affected by this flaw.

In order to avoid this bias, ISO 12571 [8] specifies two alternative methods for determining hygroscopic sorption properties of porous building materials: (i) using desiccators and weighing cups (desiccator method), considered the reference method; (ii) using a climatic chamber (climatic chamber method). The latest standard EN 16682 [13], produced in 2017, offers a wider choice of drying alternatives: (i) ventilated oven for heat resistant materials, thermostable up to $(103 \pm 2)\,^{\circ}C$; (ii) vacuum drying for materials not resistant to oven temperature, but resistant to low pressure; (iii) desiccant drying for materials neither resistant to oven temperatures nor resistant to low pressures; (iv) compressed air drying for materials neither resistant to oven temperatures nor resistant to low pressures; (v) adsorption drying for materials neither resistant to oven temperatures nor resistant to low pressures.

In wood, the gravimetric method is unaffected by pest attack, including insect tunnelling; in masonry, by the presence of salts.

2.2. Karl Fischer Titration

A chemical method, i.e. the Karl Fischer Titration (KFT) is specific to detect water in material test specimens that should be crushed to a fine powder. KFT can be performed in two basic modes. (i) Volumetric mode, where the water content of the material sample is measured with an iodine solution. This mode determines the volume of water extracted from the specimen, considering that its mass expressed in g equals its volume in cm^3. (ii) Coulometric mode, where the water content is measured with electrolysis. This mode determines the mass of water extracted from the specimen from the number of electric charges used in the electrolysis. The former mode is more robust, the latter requires smaller specimens. In both cases, the mass of the moist test specimen should be previously determined with a precision balance.

Reference should be made to the following standards

- ISO 760. *Determination of water – Karl Fischer method (General method)* [10].
- EN-ISO 15512. *Plastics – Determination of water content* [12].
- EN 16682. *Conservation of cultural heritage – Methods of measurement of moisture content, or water content, in materials constituting immovable cultural heritage* [13].

Discussion. KFT is an accurate and absolute method. EN 16682 [13] establishes that it can be used as a primary reference in addition to the gravimetric method. In the case of cultural heritage materials, KFT is limited by the ethical problem of sampling, like the gravimetric method.

In the standards [10,12], the KFT is expressed as water content of the test specimen, divided by the mass of the moist specimen. However, it is possible to convert from the moist specimen mode to the dry mode, as for the gravimetric oven drying method, and EN 16682 [13] establishes that the readings should be converted to dry, and provides the conversion formula.

In masonry, the KFT is unaffected by the presence of salts; in wood, by pest attack, including insect tunnelling.

KFT, however, might have some flaws, especially when compared with gravimetry. If the specimen includes minerals with crystallization water that is released during the titration, the method will interpret it as water content. Differently from the gravimetric method, the KFT it is not affected by oil, wax or other substances that may release VOC, because titration is specific for water. Again, oven drying is unable to extract all water molecules from the specimen, while KFT determination detects all water molecules and always yields higher moisture contents than the gravimetric method, if the two methods are expressed in the same way. Briefly, the gravimetric method and KFT extract water to a different extent, and may be affected in different way by VOCs or crystallization water. Both results are accurate, but they may be slightly different. In order to differentiate the gravimetric method from KFT, the results are differently named, i.e. "*moisture content*" and "*water content*", respectively.

2.2.1. Azeotropic distillation method

The method is based on the direct determinations of the initial mass of the moist test specimen and the mass of the water collected after distillation of the specimen. The extraction of water is made in vapour form via azeotropic distillation with a volatile solvent, immiscible with water. The water vapour is then condensed and separated in a reflux trap, recovered and measured as a volume in a graduated tube. The volume of condensed water in cm^3 numerically equals the mass in grams. The mass of the dry specimen, or the value of moisture content on the dry basis, is computed as specified for the gravimetric method. So far, the method was normalized for bitumen, i.e.:

EN 1428. *Bitumen and bituminous binders – Determination of water content in bituminous emulsions – Azeotropic distillation method* [4].

Recently, the European standard EN 16682 [13] has considered it to be used for wood.

Discussion. This is an absolute method, specific for organic materials. This method may be applied when wood contains volatile oils not soluble in water. In case the sample contains high amount of water-soluble volatile organic compounds, the level of moisture content exceeds the level of water content and a correction should be needed to remove the contribution of VOC to compare the results with other methods.

2.3. Electrical resistance method

Commercial instruments to detect the moisture content, based on the physical principle of the electrical resistance (or the electrical conductivity) between two pins pressed against the material, or inserted into it. They are very popularly used, either for wood or masonry.

A European standard has been produced for this method, but to be used strictly for wood i.e. EN 13183-2 *Moisture content of a piece of sawn timber – Part 2: Estimation by electrical resistance method* [2]. Recently, the European standard EN 16682 [13] has established limits and conditions under which the method may be used for wood and masonry too.

Discussion. This is a relative method: i.e. a measuring method whose readings cannot be expressed in terms of SI units, but in relation to something else, kept as a reference. Relative methods are especially used to detect differences over space and time.

The main advantage is that readings are provided in real time and do not require sampling, i.e. they are non-destructive, or micro-destructive when pins are inserted into the material.

The output is in arbitrary units. However, some producers calibrate the resistance output of their instruments in percent (%) unit by comparison with the gravimetric method. This scale should be, however, corrected for the specific wooden species and operating temperature. Both standards, i.e. [2,13] specify that the method should be used for normal wood, under normal physical conditions and with moisture content below the fibre saturation point. The method cannot be applied when wood has been attacked by pests (e.g. wood-boring insects, moulds) or has been previously treated with substances (e.g. oil, wax, preservatives) that alter the surface or subsurface conductivity, increasing the electrical resistance.

The method might be applied to masonry, but the presence of soluble salts increases very much the conductivity, resulting in an overestimation of the moisture content. To this aim, the British Standard BS 5250 *Code of Practise for the Control of Condensation in Buildings* [15] advices about this problem and recommends that the method shall not be used for masonry. EN 16682 [13] specifies that electrolytes in masonry do affect readings, and the method is actually expressed with (uncalibrated) relative readings. For a correct interpretation of data, the operator should specify in the test report: instrument readings, air temperature; surface temperature of the material; relative humidity of the air (all at the instant of the reading and measured following EN 15758 [16] and EN 16242 [17]). In the case of masonry, anions and cations present in the material must be determined with ion chromatography (following EN-ISO 14911 [18]; EN-ISO 10304-1 [19]) and must be specified in the test report. The operator may then evaluate the reliability of the measurement, or if it is convenient to change method. Otherwise, the method may be used for gross observations, e.g. assisting plumbers to find water leakage from pipes.

2.4. Electrical capacitance method

Commercial instruments to detect the moisture content, based on the physical principle of the electrical capacitance measurements are very popularly used, either for wood or masonry. The method is based on the physical principle that the dielectric response of materials increases in proportion to the moisture content. In fact, materials, including wood, generally have a dielectric constant ε ranging between 2 and 8, that remains (almost) unchanged, while water has a high dielectric constant, i.e. $\varepsilon = 80$ and its amount constitutes the variable.

A European standard has been produced for this method, but to be strictly used for wood, i.e.: EN 13183-3 *Moisture content of a piece of sawn timber – Part 3: Estimation by capacitance method* [3]. Later, the European standard EN 16682 [13] has established limits and conditions under which the method may be used for wood and masonry too.

Discussion. This is a relative method that may provide readings in real time, without damaging the observed material.

The output is in arbitrary units. However, some producers calibrate the output of their instruments in percent (%) but calibration is difficult and requires to operate with materials identical to those used for calibration, that is unrealistic in the case of cultural heritage materials.

The capacitance method can be applied to normal wood in good physical conditions, but it cannot be used when the wood has been attacked by pests, especially in the presence of insect tunnelling, because the loss of material will be interpreted as dryness, directly related to the proportion of material loss, with an underestimation of the moisture content. The method might be applied to masonry, but no international standards exist to encourage this method for masonry because it may lead to misinterpretation, especially in the case of deliquescent salts that reflect back the signal and the output indicates full range dampness. Physical heterogeneities or blistering may cause multiple reflections and bias, with misleadingly high dampness.

The presence of electrolytes in masonry constitutes a limit for the capacitive method too.

EN 16682 [13] norms this method too, but specifies the limitations and bias deriving when wood or masonry are investigated. In particular, for the potential presence of electrolytes it requires the additional analysis of anions and cations, determined with ion chromatography, as commented for the electrical resistance method.

3. The latest standard produced by CEN TC346 for cultural heritage materials

The European Committee for Standardization (CEN), Technical Committee for Cultural Heritage (TC346), Working Group '*Specifying and measuring indoor/outdoor climate*' (WG7) has produced specific standards for cultural heritage measurements to cope problems of aged and deteriorated materials with scientific precision and respect for artwork safety.

In particular, EN 16682 [13] is a comprehensive framework for the sustainable detection of the moisture content, or water content, in cultural heritage materials. This standard is holistic, because it norms and compares seventeen different methods, and eleven variants of them. More specifically:

- it is normative for four absolute methods and nine variants, i.e.: gravimetric with five drying options and thermogravimetry; Karl Fischer titration (volumetric-KFT, Coulometric -KFT and oven-vaporization KFT); azeotropic distillation; calcium carbide. All absolute methods shall be expressed with the same unit, i.e. % of the dry specimen;
- it is normative for three principal relative methods and two variants, i.e.: electrical resistance; capacitance; relative humidity in equilibrium with the material, either drilled-hole and external box. All relative methods are expressed in arbitrary units. Readings that have the same values, but have been taken with different methods, cannot be compared between them;
- it is informative for ten relative methods i.e.: microwave; evanescent-field dielectrometry (EFD); time-domain reflectometry (TDR); nuclear magnetic resonance (NMR); near infrared spectrometry (NIRS); ultrasound pulses; thermography; X rays; gamma rays; neutrons. It specifies their characteristics, pros and cons, as well as potential flaws.

This standard is aimed to inform and assist users with text explanations, tables, flow diagrams and decision trees. It considers and specifies characteristics, operative methodologies, discusses advantages and problems that may be met with the various methods [20,21]. It establishes a uniform presentation of data and units within each method grouping and, when possible, it gives specifications for the transformation of readings to make measurements taken with different methods comparable.

It establishes a priority of methods, i.e. first absolute, then relative methods. Among absolute methods, gravimetry and KFT have priority. Among the relative methods, electric resistance, capacitance and equilibrium relative humidity are normed. Other methods are considered not yet standardizable for normal use, because some of them require highly specialized personnel to recognize and overcome the many difficulties that may be encountered, e.g. microwave; or highly specialized instruments and personnel, i.e. NMR, X rays, Gamma rays, neutrons, NIRS, EFD, TRD; or because the method does not properly measure the moisture content, e.g. thermography.

Finally, it assists the user in the choice of the most convenient method in relation to cultural heritage materials that have undergone weathering, impregnation of oil, wax and other preservatives, pest attack, salt migration or other transformations over time, and helps to take measurements and interpret readings.

4. Conclusions

The European and international standards so far produced on moisture content [1–12] were aimed to standardize each method individually, without making comparisons. This was confusing for the user that needs to select the most convenient method for his specific case study. As opposed, EN 16682 [13], considers a wide number of methods, i.e. 17 methods plus 11 variants. Some of these methods are standardized for the first time at international level. This allows establishing an order of priority concerning accuracy and reliability.

Although this standard is specifically conceived for cultural heritage materials and deals in particular with wood and masonry, it is absolutely general and can be used for most materials, either in good or bad conditions. It cannot solve all difficulties, but warns against the most typical problems met with cultural heritage materials, or with aged and damaged materials; it friendly helps to find the most convenient method to be used, and gives advices to avoid misleading use or interpretations. For instance, the capacitive method is affected very much by voids and may be usefully employed to detect and map insect tunnelling in wooden furniture [20,21].

However, certain issues remain unresolved. In order to differentiate the gravimetric (i.e. the most popularly used) method from KFT, the results are differently named, i.e. "*moisture content*" and "*water content*", respectively. This choice of words avoids ambiguity, but there is still the matter that both methods are accurate, both can be used as primary reference for calibration, but their readings are slightly different. The user must be informed about this issue and evaluate which of them is more convenient for the specific case study.

This standard is also aimed to harmonize readings. The various methods are classified into absolute and relative. It is not possible, and it would be misleading, to use the same unit for all of them, but it has been possible to unify methods and readings within each grouping. Absolute methods (in SI units) are expressed in % by mass of the dry specimen. Others units, e.g. % by mass of the moist specimen, or % by volume, are not allowed and shall be converted. Relative methods, by their very nature, are expressed in non-comparable, arbitrary units even if tentative calibrations are made by some manufacturers to express readings in % by mass of the dry specimen. The scales applied to commercial instruments are often obscure and sometimes misleading because the responses are not linear, and the same readings may correspond to different values of moisture content. For example, the responses of a microwave instrument, a capacitive, or a resistive one are likely to differ between them considerably. A calibrated scale might constitute a reasonable approximation for some particular species of wood, in good physical conditions, if the manufacturer has specifically used the same species for calibration.

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